Paper No. 27

GAS CHROMATOGRAPHY OF CIGARETTE SMOKE, PART IV; SEPARATION ON GLASS CAPILLARY COLUMNS.

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ABSTRACT

Glass capillaries offer the following advantages over steel capillaries: As stated in part III of this study, some of the less stable smoke components do not pass steel capillaries; they are, however, detected in the effluents of glass capillaries. Retention on the glass surface is weaker than on the steel surface. Satisfying chromatograms are therefore obtained from single columns, since less rapid temperature programming is required. The low adsorption activity of the glass surface permits good separation on liquid phases of moderate polarity. The i.d. of glass capillaries drawn in our own laboratory can easily be varied and thus adjusted to particular requirements. For the analysis of fresh smoke, 0.35 mm is best suited. Preparation and coating of the glass capillaries as well as collecting of the vapor phase, and gas chromatographic techniques are described. Typical chromatograms are presented, covering, with improved separation, the same range of the vapor phase treated in part III of this investigation. Differences between results obtained from fresh and from condensed smoke are discussed.

REVIEW BY F. E. RESNIK

Dr. Grob of the University of Zurich presented a very interesting paper using glass capillary columns for the separation of cigarette smoke. He felt that a glass capillary column had an advantage over a steel capillary in that some of the less stable smoke compounds do not pass through steel capillaries and retention of these compounds on the glass surface is weaker. The technique used by Grob to coat the glass columns with a polar liquid was first to coat the capillary with a millimicron film of carbon. The polar liquid phase was then coated at a film level of 0.01 micron. Grob used a 150 meter column of 0.03 mm diameter to obtain 480,000 theoretical plates. Analysis of smoke gave 180 peaks on the glass capillary column whereas previously he had obtained 120 peaks using the steel column.

Dr. Grob emphasized the need to analyze cigarette smoke directly without collection. He felt that the cigarette smoke changes when it is allowed to stand, even at reduced temperatures. He suggested analyzing the 6th puff, since it is an average of the entire cigarette, by subjecting this puff to direct gas chromatographic separation. He is suggesting that the vapor phase smoke be handled as quickly as possible, on the order of 0.1 to 0.2 second.

REVIEW BY R. D. HELLAMS

Advantages of glass capillaries versus steel capillaries for the analysis of cigarette smoke were discussed, as indicated in the above abstract.

The liquid film of 1-2 microns thickness necessary on a glass capillary column is 20 times less thick than that necessary on steel capillaries to cover the active sites. The reduced adsorbance of glass permits the use of longer columns. The viscosity of the thinner film on the glass capillary is less important, and the use of a less polar phase on the glass capillary permits longer life of the column.

A practical rule suggested by the speaker is that if a separation is best on a steel capillary column at 150°C, then comparable work could be done at 100°C on a glass capillary column.

The disadvantage of the glass capillary is that it is a weaker adsorber than steel. A good film can be obtained with a non-polar liquid phase. The speaker noted that polar liquids form a coherent film on a glass surface which has first been covered by an approximately 0.001 μ layer of carbon black. Such pretreated glass capillaries show equal or better separation efficiencies than steel capillaries and operate at lower temperatures, due to their lower adsorption activity.

Several gas chromatograms with unlabeled peaks were shown for fresh smoke comparing the 6th and 11th puffs of a cigarette. One chromatogram appeared to be labeled as being obtained with a 100 meter capillary column of 0.32 mm ID, while the other was obtained with a 130 meter capillary column of 0.36 mm ID. These chromatograms were intended to indicate peaks seen on a glass capillary chromatogram which had not been seen - or seen only slightly - on a steel capillary chromatogram. (See part III of this investigation.)

Dr. Grob stressed the necessity of washing all tubing between gas chromatographic analyses of gas phase cigarette smoke because smoke constituents are adsorbed heavily and will influence subsequent analyses.

A specially designed 500 ml syringe connected to a Cambridge filter holder used by the speaker in this work for the collection of gas phase cigarette smoke was constructed by a Swiss watchmaker. A notched rod connected to the plunger is marked for individual puffs which are taken by hand at one minute intervals. The smoke is then pushed through the sampling valve. This method provides the advantage of analyzing the smoke from a single cigarette representing the average composition of smoke from the whole cigarette.

The use of the syringe for collecting the vapor phase of cigarette smoke from a whole cigarette is a compromise between analyzing vapor phase cigarette smoke immediately after smoking and after condensation of the smoke. Diacetyl is a possible indicator of the alteration smoke may undergo by any handling. Condensed smoke dissolved in ether and analyzed by gas chromatography showed the diacetyl had been reduced 2 1/2 times with respect to benzene.

A question from the audience concerned the sample size used in the glass capillary work for the analysis of cigarette smoke. Dr. Grob answered that this depends on the column diameter but that he used a 2 ml sample loop with a sample split of 1:50 for the work presented at this meeting.